

**WHAT IS CLAIMED IS:**

1. A method of making a sputtering target comprising the steps of:  
 providing a sputtering metal workpiece comprising at least one valve metal;  
 5 transverse cold-rolling the sputtering metal workpiece to obtain a rolled  
 workpiece; and  
 cold-working the rolled workpiece to obtain a shaped workpiece.

2. The method according to claim 1, further including the step of stress  
 relieving the sputtering metal workpiece between the steps of transverse cold-rolling and  
 10 cold-working.

3. The method according to claim 2, wherein the step of stress relieving is at  
 a temperature of from about 600 °C to about 850 °C.

4. The method of claim 1, wherein the sputtering metal workpiece is not  
 annealed or stress relieved between the steps of transverse cold-rolling and cold-working.

15 5. The method according to claim 1, further including the step of annealing  
 the sputtering metal workpiece between the steps of transverse cold-rolling and cold-  
 working.

6. The method according to claim 2, wherein the step of annealing is at a  
 temperature of from about 950 °C to about 1300 °C.

20 7. The method according to claim 3, wherein the step of stress relieving  
 comprises the step of stress relieving the sputtering metal workpiece for about 2 hours.

8. The method according to claim 6, wherein the step of annealing comprises  
 the step of annealing the sputtering metal workpiece for about 2 hours.

9. The method according to claim 1, wherein the valve metal is tantalum,  
 25 niobium, or an alloy thereof.

10. The method of claim 1, wherein said valve metal is copper.

11. The method according to claim 1, further comprising the step of machine-  
 cleaning the shaped workpiece to obtain the sputtering target.

12. The method according to claim 1, wherein the step of providing a  
 30 sputtering metal workpiece comprises the steps of:

forging flat an ingot comprising at least one valve metal;

cutting the forged ingot into slabs; and  
machine-cleaning the slabs.

13. The method according to claim 1, wherein the step of transverse cold-rolling comprises the step of cold-rolling the sputtering workpiece as a rolling workpiece  
5 a number of times in a first direction and a number of times in a second direction perpendicular to the first direction.

14. The method according to claim 1, wherein the step of transverse cold-rolling comprises the step of cold-rolling the sputtering workpiece as a rolling workpiece as many times in the first direction as in the second direction.

10 15. The method according to claim 13, wherein the step of transverse cold-rolling comprises the steps of:

cold-rolling the sputtering workpiece a number of times in the first direction, and  
thereafter cold-rolling the rolling workpiece a number of times in the second  
direction.

15 16. The method according to claim 1, wherein the rolled workpiece has a predetermined cold-rolling thickness.

17. The method according to claim 16, wherein the predetermined cold-rolling thickness is from about 0.25 inch to about 2"-gauge.

18. The method according to claim 1, further including the step of annealing  
20 the sputtering metal workpiece before the step of transverse cold-rolling.

19. The method according to claim 18, wherein the step of annealing before the step of transverse cold-rolling is at a temperature of from about 1050 °C to about 1300 °C.

20. The method according to claim 1, wherein the step of cold-working the  
25 rolled workpiece comprises deep-drawing the rolled workpiece, spin-forming the rolled workpiece, or flow forming the rolled workpiece, or combinations thereof.

21. The method of claim 1, wherein the step of cold-working the rolled workpiece comprises first deep drawing the rolled workpiece to form a preform, and then flow forming the preform over a mandrel.

30 22. The method according to claim 17, wherein the step of annealing before the step of transverse cold-rolling comprises the step of annealing the sputtering metal

workpiece for about two hours.

23. The method according to claim 1, wherein the shaped workpiece exhibits at least 50% cold reduction with respect to the rolled sputtering metal workpiece (SMW).

24. The method according to claim 1, wherein the sidewall of the shaped workpiece exhibits less than 50% cold reduction with respect to the rolled workpiece.

25. The method according to claim 1, wherein the shaped workpiece is cylindrical or cup-shaped.

26. The method according to claim 1, wherein the step of cold-working the rolled workpiece comprises deep-drawing the rolled workpiece or spin-forming the rolled workpiece, or both.

27. The method according to claim 1, further comprising the step of stress relieving the shaped workpiece after the step of cold-working.

28. The method according to claim 1, further comprising the step of annealing the shaped workpiece after the step of cold-working.

29. The method according to claim 27, wherein the step of stress relieving after the step of cold-working occurs at a temperature of from about 600 °C to about 850 °C.

30. The method according to claim 28, wherein the step of annealing after the step of cold-working occurs at a temperature of from about 900 °C to about 1300 °C.

31. The method according to claim 1, wherein the sputtering target is cup-shaped or cylindrical and has a height of about 10.5 inches, an inner diameter of about 9.25 inches, an outer diameter of about 9.50 inches and a sidewall thickness of about 0.25 inch.

32. A method of recovering valve metal from a spent sputtering target made according to the method of claim 1 comprising the step of hydriding the valve metal to obtain hydrided valve metal.

33. The method according to claim 32, further comprising the steps of:  
milling the hydrided valve metal to obtain valve metal hydrided powder;  
separating the hydrided valve metal from non-hydrided metal shell,

degassing the valve metal hydrided powder to obtain degassed valve metal

powder; and

processing the degassed valve metal powder to obtain a valve metal ingot.

34. The method of claim 1, wherein said shaped work piece has an edge, wherein said edge is subjected to cold-rolling in order to form a flange.

5 35. The method of claim 1, wherein said sputtering metal work piece is a plate and a second metal backing plate is bonded onto the first plate prior to subjecting the rolled work piece to cold-working.

36. The method of claim 35, wherein said bonding is explosive bonding, mechanical bonding, roll bonding, or combinations thereof.

10 37. The method of claim 35, wherein said second metal backing plate is copper.

38. The method of claim 35, wherein said second metal backing plate is a metal different from said sputtering metal work piece.

15 39. The method of claim 1, further comprising cutting a disc shaped work piece from said rolled work piece prior to cold-working the rolled work piece.

40. A sputtering target made according to the method of claim 1.

41. A sputtering target assembly comprising the sputtering target of claim 40, and further comprising a top portion made of a non-sputtering material attached to the sidewalls of the sputtering target, or an outer shell made of a non-sputtering material  
20 wherein the sputtering target being secured to the outer shell or both.

42. The sputtering target assembly according to claim 41, wherein the top portion is made of a valve metal base material having a strong (100) texture.

43. The sputtering target assembly according to claim 42, wherein the valve metal base material is a tantalum-base material, a niobium-base material, or both.

25 44. The sputtering target assembly according to claim 42, wherein the valve metal base material is a valve metal or alloy thereof having a strong (100) texture.

45. The sputtering target assembly according to claim 44, wherein the valve metal alloy comprises tantalum and tungsten.

30 46. The sputtering target assembly according to claim 41, wherein the top portion is made of a non-hydrating material.

47. The sputtering target assembly according to claim 41, wherein the outer

shell is made of a non-hydriding material.

48. The sputtering target assembly according to claim 47, wherein the outer shell comprises aluminum, copper, or both.

49. A target comprising at least one valve metal, wherein said target has a HCM design and said target has

- a) grain size of 5 ASTM or finer;
- b) a mixed (111)-(100) global texture;
- c) a uniform grain size wherein the grain size variance is  $\pm 2$  ASTM; or

combinations thereof.

50. The target of claim 49, wherein said target has at least two of the three properties.

51. The target of claim 49, wherein said target has all three properties.

52. The target of claim 49, wherein said target is at least partially recrystallized.

53. The target of claim 49, wherein said target is at least 95% recrystallized.

54. The target of claim 49, wherein said target is fully recrystallized.

55. The target of claim 49, wherein property a) is present and said primary (111)-type global texture is free of sharp, localized bands of (100) texture.

56. The target of claim 49, wherein property a) is present and said grain size is from about 5 ASTM to about 13 ASTM.

57. The target of claim 49, wherein property a) is present and said grain size is from about 5 ASTM to about 10 ASTM.

58. The target of claim 49, wherein property a) is present and said grain size is from about 7 ASTM to about 9 ASTM.

59. The method of claim 1, wherein said cold-working is a multi-directional cold-working.